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㉔ Sheath-core spinning of multilobal conductive core filaments.

㉕ Multilobal core conductive bicomponent sheath-core filaments are provided and methods for making the same.

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Sheath-Core Spinning of Multilobal Conductive Core Filaments

BACKGROUND OF THE INVENTION

5 Synthetic filaments having antistatic properties comprising a continuous nonconducting sheath of synthetic polymer surrounding a conductive polymeric core containing carbon black have been taught by Hull in U.S. Patent No. 3,803,453. The cross-section of the core shown in said patent is circular. Need has arisen in certain end-use applications, such as career apparel worn in clean rooms, for even greater reduction of static propensity, and contrary to the desires expressed by others to conceal the fiber blackness, is a desire for greater visibility of the core.

10 Sheath-core filaments wherein the cross-section of the core is trilobal are known. They can be prepared with a spinneret of the type shown in U.S. Patent No. 2,936,483. While useful products of the invention can be prepared with such spinnerets, improvements in preserving definition of the trilobal core through the spinning process is a worthwhile objective. The present invention offers an improved spinning technique as well as providing a novel filament which rapidly dissipates electrical charges.

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DESCRIPTION OF THE DRAWINGS

20 Figures 1 and 2 are schematic cross-sectional views of sheath-core filament of the invention illustrating trilobal and tetralobal cores as well as showing how the required structural parameters are determined.

Figure 3 is a fragmentary section of a distribution and spinneret plate taken along line 3.3 of Figure 4.
Figure 4 is a bottom view of the distribution plate of Figure 3.

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SUMMARY OF THE INVENTION

30 The present invention has two important aspects. It provides a novel synthetic filament having antistatic properties comprising a continuous nonconductive sheath of a synthetic thermoplastic fiber-forming polymer surrounding an electrically conductive polymeric core comprised of electrically conductive carbon black dispersed in a thermoplastic synthetic polymer, the cross-section of said core having from three to six lobes and a modification ratio of at least 2, with each lobe having an L/D ratio of from 1 to 20, where L is the length of a line drawn from the center point of the line between low points of adjacent valleys on either side 35 of the lobe to the farthest point on said lobe, and D is the greatest width of the lobe as measured perpendicular to L. It also provides an improved process for better maintaining the core definition during melt-spinning of a sheath-core fiber wherein one polymer composition constitutes the sheath component and a different polymer composition constitutes the core component and in which the core has three or more lobes. The process comprises simultaneously extruding the molten sheath and core component 40 compositions through a spinning orifice with the sheath component completely surrounding the core component, the improvement comprising, maintaining the core cross-sectional configuration by

- 1) feeding the molten core component composition in the desired multilobal cross-section through a channel opening above a spinneret capillary,
- 2) feeding the molten sheath component from all directions against the core along the periphery of 45 the entrance to the spinneret capillary to completely surround the core component,
- 3) controlling the flow of molten sheath component composition at spaced sections along the periphery of the spinneret capillary entrance to allow more to flow to zones between the lobes than to zones at the lobes, and
- 4) solidifying the molten components after leaving the spinneret orifice.

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DETAILED DESCRIPTION OF THE INVENTION

Static dissipating fibers are well-known in the art and have been used for many years in textiles. A

particularly successful fiber has been the fiber described in U.S. Patent No. 3,803,453. This fiber is a sheath-core bicomponent fiber prepared by melt co-extrusion of two thermoplastic compositions as sheath and core, respectively. The sheath is nonconductive. The core polymer is made conductive by incorporation of electrically conductive carbon black. The sheath provides strength to the fiber, hides the black core, and 5 protects the core against chipping and flaking which can occur if the core were exposed at the fiber surface. Certain present day end-use applications require greater anti-static effect with less concern for color. In distinction, there is a greater desire to see more core color as a means of distinguishing in use those 10 garments which are protected from those which are not. Applicants have found that this can be accomplished by modifying the sheath-core fiber of U.S. Patent No. 3,803,453. The modification consists primarily of employing a core, of the same composition as in said patent but having a cross-section with from three 15 to six lobes, a modification ratio of at least 2, and with each lobe having an L/D ratio of from 1 to 20. Figure 1 shows such a cross-section.

Figure 1 is a schematic cross-sectional representation of a sheath-core fiber wherein a trilobal core is surrounded by a sheath as might be seen on an enlargement of a photomicrograph. The nature of the core and sheath will be discussed in greater detail below. The determination of modification ratio is known in the 15 art but, for convenience, it can be defined by reference to Figure 1. The modification ratio is the ratio of the radius of the smallest circle circumscribing the trilobal core to the radius of the largest circle which can be inscribed in the trilobal core where the lobes meet. In Figure 1, this is A/B.

Determination of the L/D ratio for the lobes is also illustrated by reference to Figure 1. A first line is 20 drawn connecting the low points of adjacent valleys on either side of a lobe and another line L is drawn from the center of the first line to the farthest point of said lobe. The value D represents the greatest width of the lobe as measured perpendicular to L. Figure 2 is a schematic showing a cross-section of a round fiber having a tetralobal core.

Spinning of the filaments of the invention can be accomplished by conventional two-polymer sheath- 25 core spinning equipment with appropriate consideration for the differing properties of the two components. The filaments are readily prepared by known spinning techniques and with polymers as taught, for example, in U.S. Patent No. 2,936,482. Additional teaching of such spinning with polyamides is found in U.S. Patent No. 2,989,798. A new improved process has been developed to better preserve the definition of sheath-core 30 bicomponent fibers having tri-, tetra-, penta- or hexalobal cores as they are extruded. This is described below.

The improved process employed for spinning the sheath-core bicomponent yarn of Examples 1 and 2 below, is a modification of a conventional sheath-core bicomponent melt-spinning process. In the conventional process, the core feed polymer stream and the sheath feed polymer stream are fed to a spinneret pack including filters and screens, and to a plate which distributes the molten polymer streams to orifices 35 that shape the core and surround it with sheath. Reference to Figures 3 and 4 will assist in the understanding of the modified process. Core polymer is fed to channel 2 and exits over the entrance to capillary 3 of spinneret plate 5. Sheath polymer is fed through passageway 7 of plate 8 into the space between plates 5 and 8, maintained by shims not shown. This polymer is fed from all directions against the core polymer stream in the vicinity of the entrance to the spinneret capillary 3 and both streams pass 40 through capillary 3 in sheath-core relation, finally exiting from the spinneret orifice, not shown, at the exit of capillary 3. The improved process maintains better definition of the core lobes. This is accomplished by controlling the flow of molten sheath component composition against the core polymer stream at spaced sections along the periphery of the entrance to the capillary to allow more sheath polymer to flow to zones between the lobes than to zones at the lobes. This can be achieved by enlarging the passageway for the 45 sheath polymer to the capillary only in those sections leading to zones between lobes. Thus, as shown in Figures 3 and 4, depressions 10 were etched in plate 8 to permit increased sheath polymer flow to regions between lobes.

The filament sheath may consist of any extrudable, synthetic, thermoplastic, fiber-forming polymer or 50 copolymer. This includes polyolefins, such as polyethylene and polypropylene, polyacrylics, polyamides and polyesters of fiber-forming molecular weight. Particularly suitable sheath polymers are polyhexamethylene adipamide, polycaprolactam, and polyethylene terephthalate.

Tensile and other physical properties of the filaments of the invention are primarily dependent on the 55 sheath polymer. For high strength filaments, polymers of higher molecular weight and those permitting higher draw ratios are used in the sheath. While undrawn filaments of the invention may provide adequate strength for some purposes, the drawn filaments are preferred. In some applications, for example where the filaments of the invention are to be subjected to high temperature processing with other filaments such as in hot fluid jet bulking or other texturing operations, it is important that the sheath polymer have a sufficiently high melting point to avoid undue softening or melting under such conditions.

The filament core of the antistatic fibers consists of an electrically conductive carbon black dispersed in a polymeric, thermoplastic matrix material. The core material is selected with primary consideration for conductivity and processability as described in detail in U.S. Patent No. 3,803,453. Carbon black concentrations in the core of 15 to 50 percent may be employed. It is found that 20 to 35 percent provides the 5 preferred level of high conductivity while retaining a reasonable level of processability.

The core polymer may also be selected from the same group as that for the sheath, or it may be non-fiber forming, since it is protected by the sheath. In the case of non-antistatic fibers, the core of the bicomponent fiber will, of course, be non-conductive.

The cross-sectional area of the core in the composite filament need only be sufficient to impart the 10 desired antistatic properties thereto and may be as low as 0.3 percent, preferably at least 0.5 percent and up to 35 percent, by volume. The lower limit is governed primarily by the capability of manufacturing sheath/core filaments of sufficiently uniform quality while maintaining adequate core continuity at the low core volume levels.

Conventional drawing processes for the filaments can be used but care should be exercised to avoid 15 sharp corners which tend to break or damage the core of the antistatic fibers. In general, hot drawing, i.e., where some auxiliary filament heating is employed during drawing, is preferred. This tends to soften the core material further and aid in drawing of the filaments. These antistatic filaments may be plied with conventional synthetic, undrawn filaments and codrawn.

For general applications, the filaments of this invention have a denier per filament (dpf) of less than and 20 preferably less than 25 dpf.

The filaments of this invention are capable of providing excellent static protection in all types of textile end uses, including knitted, tufted, woven and nonwoven textiles. They may contain conventional additives and stabilizers such as dyes and antioxidants. They may be subjected to all types of textile processing including crimping, texturing, scouring, bleaching, etc. They may be combined with staple or filament yarns 25 and used as staple fibers or as continuous filaments.

Said filaments may be combined with other filaments or fibers during any appropriate step in yarn production (e.g., spinning, drawing, texturing, plying, rewinding, yarn spinning), or during fabric manufacture. Care should be taken to minimize undesirable breaking of the antistatic filaments in these operations.

Upon exiting the spinneret orifice, the bicomponent stream cools and begins to solidify. It is generally 30 not desirable to apply too high a spin stretch with the conductive fibers since quality as an antistatic fiber diminishes. This is not a limitation with other bicomponent fibers.

TEST PROCEDURES

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Tenacity and elongation of yarns were measured using ASTM D-2256-80. The method for determining relative viscosity (LRV) of polyester polymers is described in U.S. 4,444,710 (Most). The method for determining relative viscosity (RV) of polyamides is disclosed in U.S. 4,145,473 (Samuelson). Surface 40 resistivity of fabrics is determined using AATCC Test Method 76-1987. Electrostatic propensity of carpets is measured using AATCC Test Method 134-1986. Static decay data are measured using Method 4046 (March 13, 1980), Federal Test Method Std. No. 101C. The modification ratios and L/D ratios were measured from cross-sections on photomicrographs as well understood in the art.

The following examples, except for controls, are intended to illustrate the invention and are not to be 45 construed as limiting. Multilobal core filaments of the invention are described in each of Examples 1 to 3.

EXAMPLE 1

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Sheath-core filaments having a sheath of 23.5 LRV polyethylene terephthalate and a polyethylene core that contained 28.4% carbon black were spun and wound up without drawing at 1200 meters per minute. The conductive core constituted 6% by weight of these filaments, and the yarns, which contained six filaments, were subsequently heated to 140 °C and drawn at the ratios listed in Table I. Samples with a 55 round conductive core were spun using a spinneret assembly similar to that shown in Figure 11 of U.S. 2,936,482, whereas those having trilobal shaped cores were spun by the improved process of this invention using the spinneret assembly and plate shown in Figures 3 and 4. The modification ratio of the trilobal conductive core was 5 and the L/D ratio was 3. The trilobal core yarns were darker than the round core

yarns. After drawing, these yarns were incorporated into a 100% polyester 28 cut jersey knit by feeding in the conductive core yarns at 5/16 inch intervals. Yarn and fabric properties measured on these samples are shown in Table I:

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Table I

10	Core Shape	Round	Trilobal
	Draw Ratio	2.35X	2.10X
	Total Denier	35.9	40.0
	Tenacity, g/d	1.81	1.61
	% Elongation	28.9	21.4
15	Fabric Properties		
	Surface Resistivity ohms/unit sq.	1.5 X 10 ¹³	1.9 X 10 ¹²
20	Federal Test Method 4046		
	Standard 101c (90% Decay)		
25	Time in sec./2 sec. charge level		
	From:	+ 5KV -5KV	33/900 9.5/-950
			0.23/275 0.20/-300

The fabric containing the yarn with the trilobal shaped conductive core had significantly lower surface resistivity and much faster static decay times than that made with the yarns having round conductive cores.

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EXAMPLE 2

35 Sheath-core filamentary yarns (40 denier 6 filaments) having a sheath of 46 RV 66-nylon and either
36 round or trilobal shaped conductive cores similar to those described in EXAMPLE 1 were prepared, except
37 they were drawn at 110 °C using a 3.2X draw ratio. The modification ratio of the trilobal conductive core
38 was 4 and the L/D ratio was 2. These conductive core fibers were plied with 1225 denier nylon carpet yarn
39 and direct tufted into level loop carpets. Both carpets were evaluated in the AATCC Test Method 134. The
40 carpet containing the yarns with trilobal shaped cores had a significantly lower measurement of 0.8 KV
versus 1.2 KV for the carpet made from yarns having round conductive cores.

EXAMPLE 3

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Utilizing spinneret assemblies as described in Figure 11 of U.S. 2,936,483, sheath-core products were produced having a 24% central conductive core surrounded by a 76% sheath of polyethylene terephthalate. Filaments having either round or trilobal (modification ratio of 2.0, L/D of 1.0) shaped conductive cores were prepared, and the cores contained 32.0% carbon black ("Vulcan P", available from Cabot Corp.), compounded into a film grade equivalent high melt index, low density polyethylene.

The resulting fibers were air quenched at 21 °C, drawn 1.84X and wound up at 1372 meters per minute as a 35 denier 6 filament product. After heat annealing (130 °C) to reduce shrinkage, the products were woven into fabric for static dissipation evaluation.

55 Woven fabrics were prepared as follows:

Non-Conductive Yarns - 150 denier, 34 filaments - 3.3Z twist polyester fiber.

Non-Conductive Yarns - 150 denier, 34 filaments - 3.82 twist polyester fiber
Static Dissipative Yarns - 100 denier, 34 filaments - 4S twist polyester fiber plus one static dissipative yarn
as described above

Weaving:

96 ends, 88 picks, 8 x 8 herringbone

Warp - 1 Static dissipative yarn and 23 non-conductive ends.

Filling - 2 Static dissipative yarns and 22 non-conductive picks.

5 Fabrics:

- A. Contains Trilobal Core
- B. Contains Round Core Electrostatic Properties

Yarn Resistivity, ohms/cm (length)-as prepared.

10 A. 3.7×10^{11}
 B. 7.4×10^{11} Fabric Resistivity (AATCC 76-1987) ohms/unit square after heat-setting and scouring.
 A. warp- 2.9×10^{12} , fill- 2.7×10^{12}
 B. warp-> 1×10^{14} , fill-> 1×10^{13}

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Claims

1. A novel synthetic sheath-core bicomponent filament having antistatic properties comprising a continuous nonconductive sheath of a synthetic thermoplastic fiber forming polymer surrounding an electrically conductive polymeric core comprised of electrically conductive carbon black dispersed in a thermoplastic synthetic polymer, the cross-section of said core having from three to six lobes and modification ratio of at least 2, with each lobe having an L/D ratio of from 1 to 20, where L is the length of a line drawn from the center point of the line between low points of adjacent valleys on either side of the lobe to the farthest point on said lobe, and D is the greatest width of the lobe as measured perpendicular to L.
2. A filament in accordance with claim 1 wherein the core constitutes at least 0.3% of the filament cross-section.
3. A filament in accordance with claim 1 wherein the core constitutes up to 35% of the filament cross-section.
4. A filament in accordance with claim 1 wherein the sheath component is a polyester.
5. A filament in accordance with claim 1 wherein the sheath component is a polyamide.
6. A filament in accordance with claim 1 wherein the core polymer is polyethylene.
7. A fabric containing the filament of claim 1.
8. A carpet containing the filament of claim 1.
9. In a process for melt-spinning a sheath-core fiber wherein one polymer composition constitutes the sheath component and a different polymer composition constitutes the core component and the core cross-section has three or more lobes, which process comprises simultaneously extruding both molten component compositions, through a spinning orifice with the sheath component completely surrounding the core component, the improvement comprising, maintaining the core cross-sectional configuration by
 - 1) feeding the molten core component composition in the desired multilobal cross-section through a channel opening above a spinneret capillary,
 - 2) feeding the molten sheath component from all directions against the core along the periphery of the entrance to the spinneret capillary to completely surround the core component,
 - 3) controlling the flow of molten sheath component composition at spaced sections along the periphery of the spinneret capillary entrance to allow more to flow to zones between the lobes than to zones at the lobes, and
 - 4) solidifying the molten components after leaving the spinneret orifice.
10. A process according to claim 9 wherein the core component composition is electrically conductive.
11. A process according to claim 10 wherein the core component comprises electrically conductive carbon black dispersed in polyethylene.

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FIG. 1

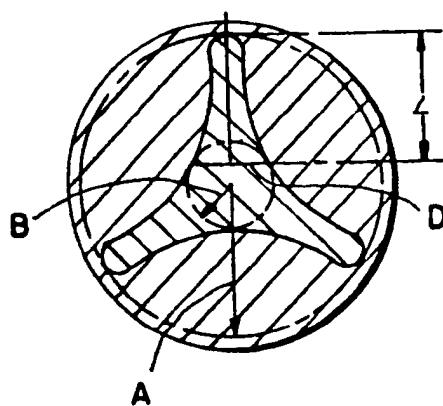


FIG. 2

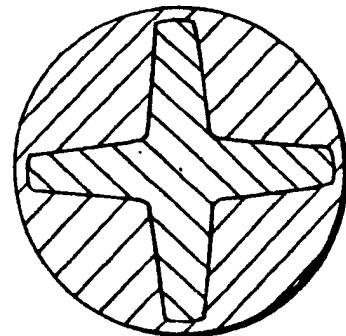


FIG. 3

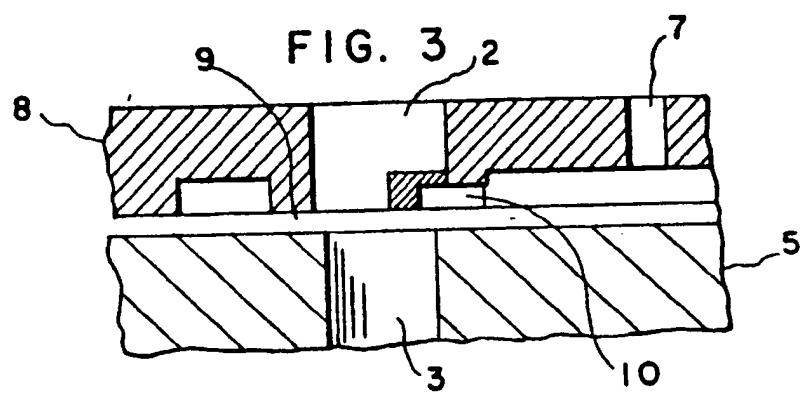
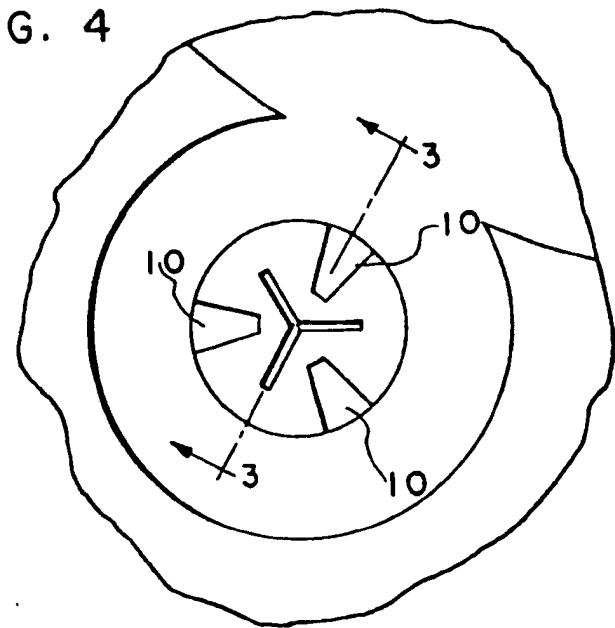


FIG. 4







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(54) Sheath-core spinning of multilobal conductive core filaments.

(57) Multilobal core conductive bicomponent sheath-core filaments are provided and methods for making the same.

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REPORT

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DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int. Cl.5)		
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim			
P,Y	PATENT ABSTRACTS OF JAPAN, vol. 13, no. 472 (C-647)[3820], 25th October 1989; & JP-A-1 183 519 (TEIJIN LTD) 21-07-1989 * Whole document * - - -	1-4,6-8	D 01 D 5/253 D 01 D 5/34 D 01 F 1/09		
Y	PATENT ABSTRACTS OF JAPAN, vol. 13, no. 15 (C-559)[3363], 13th January 1989; & JP-A-63 219 627 (TEIJIN LTD) 13-09-1988 * Whole document * - - -	1-4,6-8			
Y	US-A-3 568 249 (MASAO MATSUI) * Abstract * - - -	1-11			
D,Y	US-A-3 803 453 (D.R. HULL) * Claims 1,2,8,9,10 * - - -	1-11			
A	US-A-4 207 376 (T. NAGAYASU et al.) - - - -				
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)		
			D 01 D D 01 F		
The present search report has been drawn up for all claims					
Place of search	Date of completion of search	Examiner			
The Hague	19 March 91	TARRIDA TORRELL J.B.			
CATEGORY OF CITED DOCUMENTS					
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